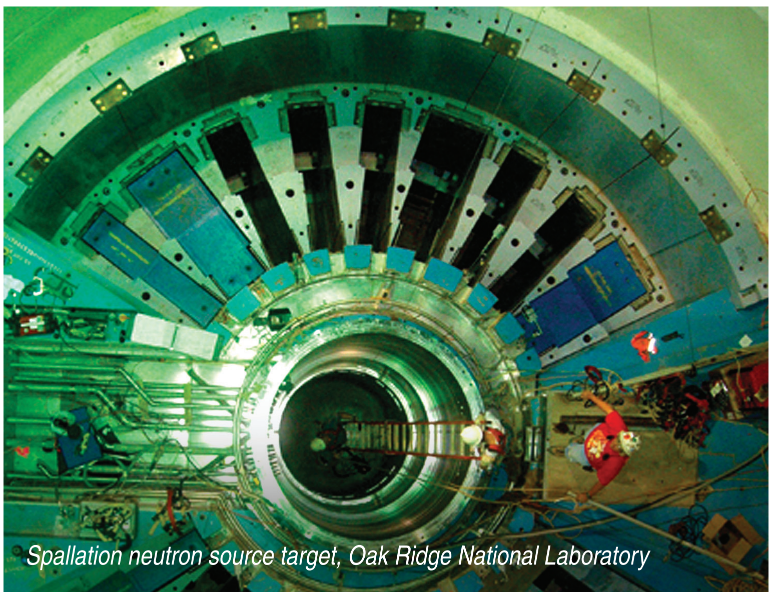
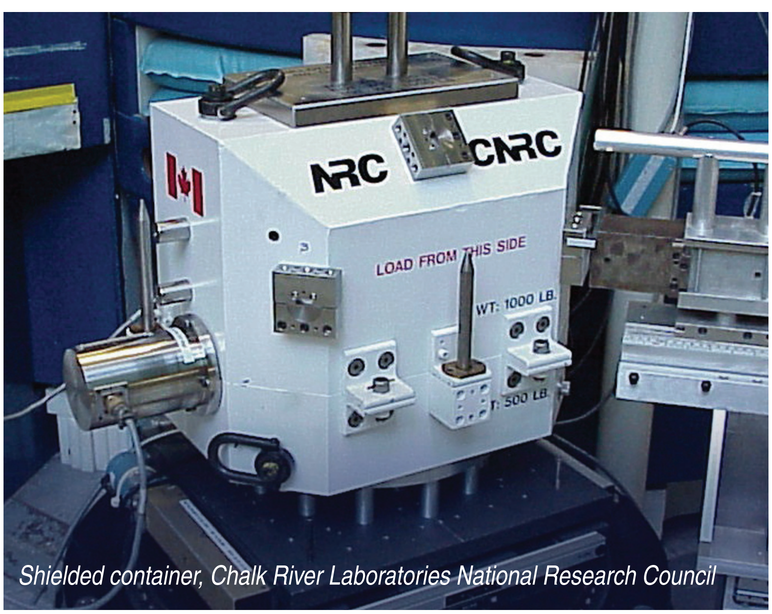
Advanced photon source target, Argonne National Laboratory



Spallation neutron source target, Oak Ridge National Laboratory



Shielded container, Chalk River Laboratories National Research Council

Research Needs and Opportunities for Characterization of Activated Samples at X-Ray and Neutron User Facilities

Bishops Lodge, Santa Fe, NM
September 20—22, 2009

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Attendees at the workshop, Research Needs and Opportunities for Characterization of Activated Samples at X-Ray and Neutron User Facilities, held in Santa Fe, New Mexico.

Left to right: Thomas Proffen, Phil Withers, and Lyndon Edwards

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Table of Contents

1. Executive Summary

On September 20-22, 2009, a workshop entitled Research Needs and Opportunities for Characterization of Activated Samples at X-Ray and Neutron User Facilities, sponsored by Los Alamos National Laboratory, was held in Santa Fe, New Mexico. The workshop was motivated by the potential that light and neutron sources have to proffer advances in our understanding of radiation damage and to validate new science-based materials performance models. The charge of the workshop addressed measurement needs, current activity, and opportunities that can be realized in the next five years and insights that could be realized by new diagnostics and experimental methods. This report outlines the content and discussion of the workshop.

Discussions of the anticipated need covered fuels, structural applications, and materials discovery for fission and fusion reactors. For fuels priority, needs included characterization of defect distributions, voids, bubbles, cracks, precipitates, new chemical phases, alloy species redistribution, recrystallization, and grain growth. For structural components, priorities included the improved understanding of the changes in yield, ultimate tensile strength, embrittlement, and fracture toughness. Radiation-assisted stress corrosion cracking was also identified as very important. For new materials, an improved understanding of the interactions between strengthening features and radiation-induced defects was considered crucial.

When considering the opportunities available at existing facilities, the provision of three-dimensional spatial distributions of defect and chemical distributions with atomistic resolution were areas of opportunity, especially if they have temporal resolution consistent with the phenomena of interest. However, realizing the full potential of user facilities will, in many cases, require changes in the infrastructure and the requirements that are necessary for safe and efficient handling of activated material. Moreover, facilities need to provide sample preparation areas and bring to bear the full range of diffraction and spectroscopic techniques on increasingly small (and thus more radiologically manageable) samples.

Plausible priority research opportunities identified for the next five years included the following: 1. In situ crystallographic response to applied stress in archived irradiated materials; 2. Crack growth under fatigue conditions in irradiated alloys (e.g. zirconium) containing hydrides; and 3. Small angle scattering and diffraction of precipitates (e.g. M23C6 particles in Fe9%Cr steels) to explore the formation and strain fields around deformation-induced voids.

In workshop discussions concerning a decadal future facility, numerous opportunities were identified that coupled X-ray or neutron probes with an irradiation source: examination of individual grains in activated samples; handling and characterization of "large" components; in situ measurement of creep properties in conjunction with radiation and helium ingress; and defect kinetics measurements and characterization of spent fuel. No less important are handling facilities and hot cells. The requirement to have all the classical post-irradiation microscopy, such as electron microscopy and ion beam facilities, was integral to the vision. On the decadal time scale, the following areas of interest were identified of special import: 1. Materials for Generation 4 reactors; 2. In situ fatigue testing at temperatures that allow the quantification of fatigue-irradiation-creep interaction; and 3. Examination of individual particles to see how these interact under deformation with and without irradiation.

If the promise of the so-called nuclear renaissance is to be realized, especially in the United States, the breadth and depth of the nuclear science and engineering community must be enhanced substantially. In particular, there is a need to revitalize the materials science of radiation damage. It was clear to workshop participants that x-ray and neutron sources at national user facilities have an important role to play in this endeavor. Further, in addition to cultural changes that would allow the full exploitation of currently available tools and techniques, new capabilities need to be developed if science-based certification is to play a role in the resurgence of nuclear energy. Finally, given the magnitude and urgency of the need for carbon-neutral energy, approaches must be found to reduce the time and cost associated with licensing and certification.

2. Introduction

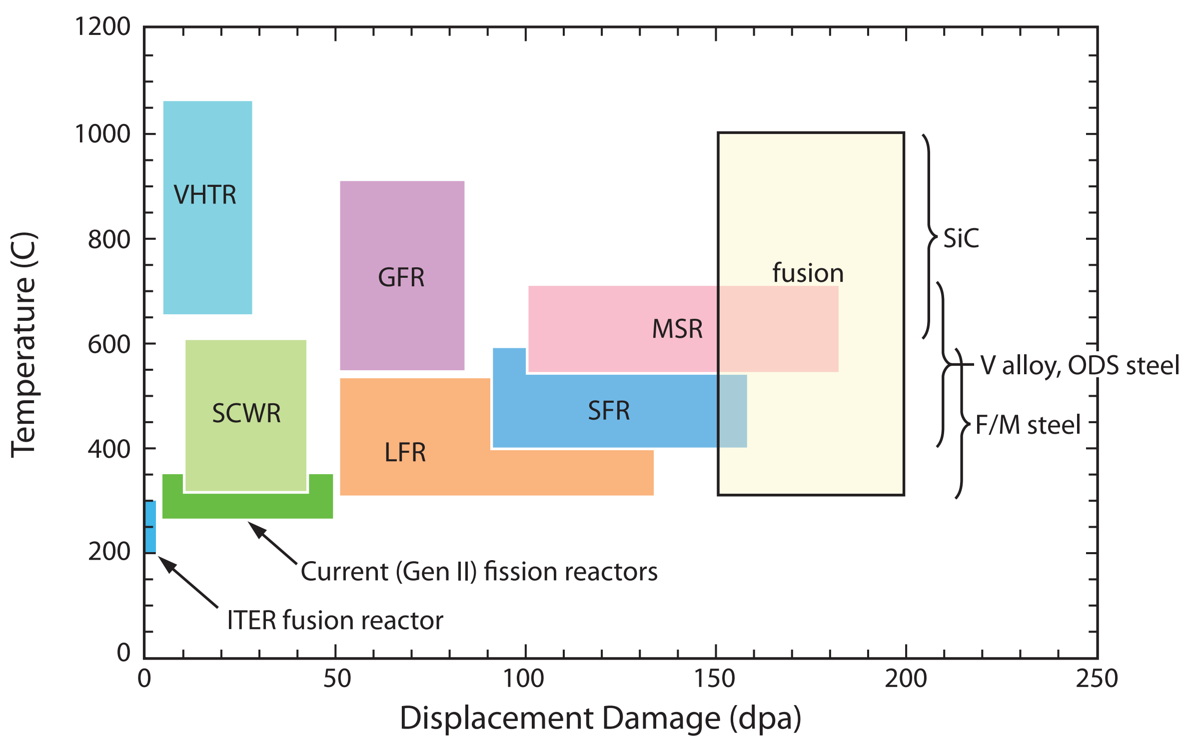
The current generation of nuclear power reactors was designed nearly 50 years ago and since then dramatic advances in tools for the pursuit of materials science have been realized. These include super-computers, X-ray and neutron user facilities. (See “Neutrons and Synchrotron Radiation in Engineering Materials Science” edited by W. Reimers, A.R. Pyzalla, A. Schreyer and H. Clemens, Wiley-VCH Verlag GmbH & Co. KGaA, Wertheim, 2008.) Using these tools, it seems likely that breakthrough insights will be possible in the next decade concerning the behavior of materials exposed to radiation damage.

The field of radiation damage research is heavily influenced by its relevance to the safety case of engineering applications in fission power generation. Much of the research and development is directed at this end-use. Current fission reactors present many managed but poorly understood problems, concerning the strength and practical life of materials. Material selection in fission power applications is far from a mature technology. As increased burn-up, fluence and temperatures are considered for fission and fusion applications, the need for improved understanding is compelling (see Figure 1). The potential societal implications are considerable. For example in the United States alone, the question of whether lifetime extensions are granted to the existing fleet of nuclear power stations has a fiscal import of billions of dollars.

The origin of radiation damage begins at the atomic scale but the accumulation and interaction of radiation-induced defects compromises macroscale properties. Ultimately, the engineering performance of fuels or structural materials in a reactor depends on these defects and, in particular, on their interaction with the microstructure. At the atomic level, the problems are as fundamental as any found in materials science. However, extrapolating from atomistic insights to macroscopic length scales requires a complex and difficult synthesis of science, metallurgy and engineering.

This workshop was motivated by the potential that new tools at light and neutron sources have to proffer unique advances in our understanding of radiation damage. These tools have the potential to inform and validate atomistic codes in ways

Figure 1. After S.J. Zinkle ,OECD NEA Workshop on Structural Materials for Innovative Nuclear Energy Systems, Karlsruhe, Germany, June 2007, in press



that have been hitherto impossible. With wider application, the resulting insights might accelerate our understanding and certification of materials used in nuclear applications. A subordinate motivation was a desire to explore opportunities that would complement Los Alamos National Laboratory's Matter-Radiation Interactions in Extremes (MaRIE) signature facility concept. The workshop was preceded by three Office of Science workshops: Basic Research Needs for Advanced Nuclear Energy Systems, Basic Research Needs for Materials Under Extreme Environments, and Next Generation Photon Sources.

The workshop was held Sept. 20-22, 2009, and focused on user facilities. The charge addressed the science and engineering challenges that warrant examination, current activity, opportunities that can be realized in the next few years and insights that could be realized by new diagnostics and experimental methods. Attendees came from many of the U.S. national laboratories, including Los Alamos, Lawrence Livermore, Argonne, Oak Ridge, Pacific Northwest and Idaho. U.S. industry was represented by two attendees from the Electric Power Research Institute and a representative from the Nuclear Regulatory Commission. There were six attendees from U.S. universities and six international attendees.

3. Measurement Needs

3.1 Introduction

The range of irradiated and activated samples that can benefit from examination at user facilities is considerable. Opportunities range from fundamental studies to practical studies. Fuels and structural materials were a common engineering theme over a wide range of dose, rate and temperature conditions. Some problems such as leaking fuel pool liners are of immediate import while others such as designs for a conceptual fusion reactor first wall have a much longer lead time. Noting the current advocacy for greater impact of modeling on certification and discovery, the workshop focused on tools that would contribute to the perennial desire to link models over multiple length and temporal scales.

In hot cells, characterization tools typically span the range of lab scale techniques such as electron microscopy, Auger Atom probe spectroscopy, positron annihilation spectroscopy, Raman, mechanical testing, etc. Often the studied materials are surveillance coupons, and the properties of interest are those most pertinent to engineering: hardness, tensile properties, toughness, residual stress, texture, yield strength, strain hardening, corrosion and oxidation rates, and materials compatibility. Handling irradiated samples in hot cells with remote handling is routine if not cheap. Whereas x-ray and neutron user facilities often provide opportunities to examine a wider range of phenomena non-destructively on smaller samples with greater precision, spatial, and temporal resolution, this capability is sparsely applied in no small part because of the reticence and lack of infrastructure at most user facilities to handle highly activated materials. Nevertheless, there are counter examples such as the Stanford Synchrotron Radiation Lightsource, which accepts samples up to 10 GBq (as indeed will the MARS soleil facility in France) or the Chalk River facility where shielded containers have been used to make neutron diffraction measurements on samples up to 20,000 R/hour.

Listed below are three problem areas that could strongly benefit from increased measurements: nuclear fuels, structural components, and new materials.

3.2 Nuclear Fuels

UO2 and mixed (uranium and plutonium) oxide are the primary candidates for fuels for new reactors over the next 25 years. The factors limiting fuel performance are the defect distributions, voids, bubbles, cracks, precipitates, new chemical phases, alloy species redistribution, recrystallization, and grain growth generated by radiation. Typically, information on these factors is needed as a function of radial position from the centerline to the fuel cladding and has been expensively obtained by classical microscopy techniques, but new methods may allow it to be determined non-destructively.

One of the potential advantages of fast-breeder fuels is their potential to offer higher burn-up when compared to current nuclear fuels (10% versus 3%). One candidate design is tristructural-isotropic (TRISO) fuel which comprises spheres (mm diameter) of (e.g.) Uranium carbide surrounded by moderating layers of carbon, contained within a stainless steel cladding. The result is a fuel that is inhomogeneous on the scale of the size of the spheres. Therefore, its behavior will be locally inhomogeneous on a similar length scale. Since it is expected to operate at higher temperatures, perhaps using liquid metal coolants, experimental capabilities are urgently needed to follow the inhomogeneous formation of defects, voids, crack, bubbles, and phase-changes that will form during irradiation. Fuel deterioration due to cracking of fuel within the cladding is one phenomenon that decreases the effective thermal conductivity and limits the rating of the fuel. Thus, a major advance in our understanding of the life limiting processes could be achieved if it were possible to follow fuel damage in situ. This is true for regular oxide fuels but will be even more important where the swelling will be exacerbated in fast reactor fuels.

There is also a need to monitor waste-form stability in which the evolution of new phases and damage accumulation contributes to the degradation of leachability barriers. For example the α-decay of irradiated UO2 causes radiolysis of water. The ensuing chemical reaction changes U4+ ions to U6+ ions, which are soluble in water, permitting uranium to be leached out of the waste. Adding H+ ions to the water produces a reducing environment that blocks the U4+ to U6+ pathway. How this occurs is not understood. With neutron reflectometry, it should be possible to examine the surface behavior of UO2 using D2O/H2O contrast matching to nullify the water scattering.

3.3 Structural Components

Understanding the degradation of the mechanical properties of the structural materials used within a reactor is vital to its operating safety case. In a radiation environment changes in yield and ultimate tensile strength, embrittlement, and loss of fracture toughness are all common. The fracture toughness, yield-point, and temperature determine what length a crack has to be before it grows rapidly is reasonably well understood in most cases. However less well understood is the effect of irradiation on fatigue (the response to cyclic loads provided by temperature cycling or vibrations caused by water flow), creep (the gradual growth of structures at high temperature and under stress), and their interaction.

Figure 2. A 7.5-inch diameter nuclear power plant pressurizer nozzle (after David Rudland)

Residual stresses associated, for example, with girth welds in nuclear pipe-work (such as nozzles close to the reactor pressure vessel) are important since a weld failure could result in a loss of coolant event (e.g., Radiation-induced stress relaxation of welded type 304 stainless steel," M. Obata, J.H. Root, Y. Ishiyama, K. Nakata, H. Sakamoto, H. Anzai, and K. Asano Proc 22nd Symp. on effects of radiation on Materials, ASTM STP1475, p. 15, Boston 2004). Since the magnitudes of the residual stresses generated by welding are often unknown, their assumed values often play a more important role in limiting lifetimes than the in-service stresses. In order to obtain a damage-tolerant estimate of remaining life, some assumption or knowledge about these residual stresses is needed to model crack propagation through welded material whose toughness has been reduced by radiation. Thus, radiation-assisted stress corrosion cracking is very important to both boiling water and pressurized water reactors. The Electric Power Research Institute (EPRI) has major national and international programs to investigate stress corrosion cracking particularly in the context of modern automated welding practices when compared to manual methods used in the past. The welds of interest are often complicated since they join body-centered cubic pressure vessel steels to corrosion resistant stainless steel and are sometimes thick, involving 3-6in. piping in which the welds are highly constrained by the pipe thickness (see fig 2).

Another important aspect of reactor safety is the integrity of the fuel cladding against breakage and the potential release of fuel and fission products resulting from cladding failure is of considerable importance. For zircaloy cladding, the issues are corrosion, hydride accumulation, growth driven by intergranular strains and crystallographic texture, and exacerbation by stress fields induced by manufacture or welding. Cladding integrity will be of even greater concern in future fast reactor systems for which potentially increased burn-up leads to greater swelling and hence pressure on the cladding.

3.4 New Materials

New materials of possible relevance for structural applications have recently been identified from research on so-called Generation 3 and 4 materials. These include oxide dispersion strengthened steels, ferrite/bainite (P91) alloys, and alloys of the form Ti3AlC2. In many cases, they appear to have good strength and radiation resistance but have little or no operational record in representative extreme radiation environments. Thus, a scientific understanding of the interactions between strengthening features and the radiation-induced defects is of paramount importance if they are to be certified for new applications. Segregation of alloying elements and the role of interfaces are two important areas of interest. Another need is the possibility of improving current materials within the envelope of their certification to avoid the drawn-out process of certifying anew.

Recent studies have shown some materials that have superior resistance to radiation, for instance the layered material CuNb, may attain this by virtue of the interfaces. Materials with strengthening features at the scale of nm often have superior mechanical properties. The reason for this improvement in properties may well be what happens in the interfaces rather than within the grains. Cladding material shows void denuding near the grain boundaries, suggesting that the interfaces are sinks for defects. If this proves to be the key phenomenon then diffraction (which originates in coherent effects in the grains), may be a less valuable probe than one which is sensitive to conditions in grain boundaries. Fortunately, the size of synchrotron beams is becoming smaller while still retaining intensity. Within the next decade, it may be possible to perform diffuse scattering from interfaces such as grain boundaries.

4. Near-Term Opportunities (One to Five Years)

4.1 Methods for Transformational Insights

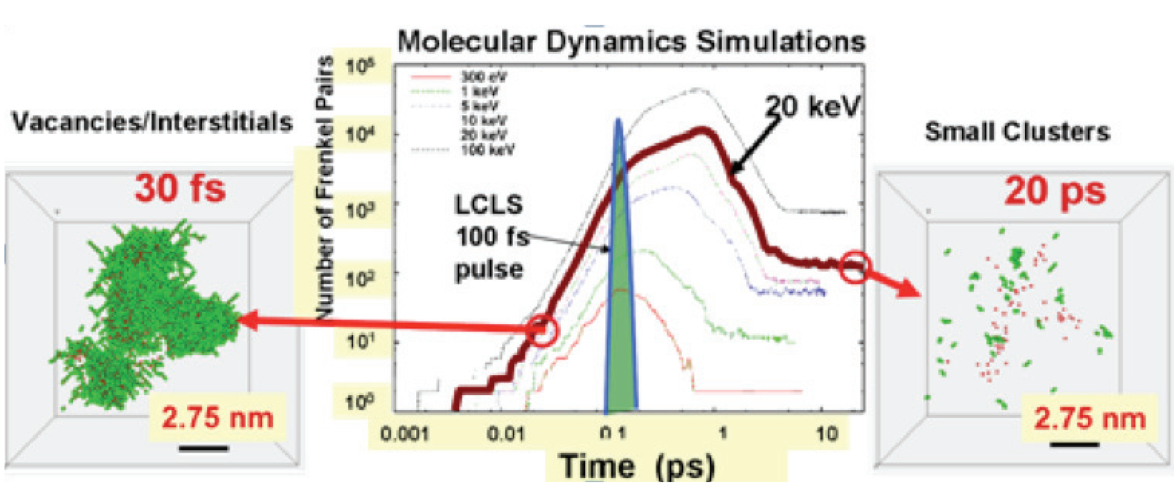
Much of what we think we know about primary damage formation comes from molecular dynamics (MD) simulations. However, much experimental effort misses the spatial and temporal regimes most pertinent to MD by focusing on long range or bulk average phenomena, such as resistivity, temperature, swelling, constitutive response and corrosion rates. Thus, new tools that provide three-dimensional spatial distributions of defect and chemical distributions with atomistic resolution could prove invaluable, especially if they have temporal resolution consistent with the phenomena of interest.

There is also a compelling need for engineering studies using new probes that can operate under extreme irradiation environments. For fuels, issues of interest include melt temperature as a function of actinide composition and chemistry; dimensional stability, thermal properties, and material diffusion as a function of chemistry, temperature and microstructure; heat generation from nuclear processes; fission product accumulation and gas release. Issues for cladding include strength and ductility as a function of microstructure, temperature, and chemistry; actinide and fission product diffusion; and chemical reactions at fuel-clad interface. In either case, the potential to make measurements during simulated failures such as loss-of-coolant accidents would allow failure margins to be explored.

Fig 3. Defect production during radiation (after Malcolm Stocks)

Developments at third and fourth generation light sources as well as at new neutron sources such as the spallation neutron source at Oak ridge national laboratory hold the potential for unprecedented insights into fundamental processes that dictate radiation damage. Several specific areas of immediate opportunity were identified: diffraction measurements during loading of archival samples; residual stress measurements of structural welds; property determination of irradiated oxide dispersion strengthened/nano structured ferritic steels; inelastic neutron scattering on samples of atypical isotopic composition (e.g., Pu 242); resonant inelastic X-ray scattering fluorescence measurements (e.g., on Americium at high pressure) or elemental mapping on the sub-micron scale (0.03 µm resolution) with a sensitivity to chemical species of parts per billion; and development of advanced x-ray and neutron focusing optics (e.g., for studies of ion beam irradiated layers).

Opportunities implicit in the Linac Coherent Light Source (LCLS) were a frequent focus. The potential of LCLS to observe with pico-second resolution, defects, their interactions and dynamics may lead to better charting of damage pathways (see fig 3). The high intensity short photon pulses offer experimental potential that, for the first time, complements high performance computing atomistic models at the shortest relevant spatial and temporal scales. Realizing this potential will require the development of techniques that can use such radiation sources to measure nucleation of defect clusters, bubbles/voids, image dislocations, grain boundaries, and precipitates, and demonstrate the interaction of dislocations with defect clusters, bubbles, and voids.



4.2 Radiography and Tomography

X-ray tomography can be pursued using laboratory or synchrotron sources that complement one another by investigating components over a range of length scales. Laboratory tomography can effectively cover the spatial range of 1-10 μm taking between a half hour and 10 hours for a scan. It has been applied to components of the order of a millimeter in maximum dimension such as crack growth in nuclear graphite, damage in LiTiO3 "pebbles" for fusion blankets and TRISO fuel elements. Defects in the SiC coating around a TRISO particle can readily be resolved. Conversely synchrotron tomography, carried out at major user facilities, and therefore requiring more organization, covers the size range 0.2-1.0 μm with scan times ranging between a few seconds and an hour. (High energy microtomography, with a resolution of 0.2 μm in a few seconds, has been applied to viewing crack growth in compact tension samples in real time.) Applications to visualizing defects in an irradiated microstructure are easy to envision.

Neutron tomography with a resolution of 200 μm continues to be limited by the availability of high resolution, two-dimensional detectors and by flux. Nevertheless new facilities are being built in Australia for examination of radioactive components, and new general user facilities are being built at the ISIS neutron source in the United Kingdom and at the Spallation Neutron Source at Oak Ridge National Laboratory. The continuous spallation source at the Paul Scherrer Institute in Switzerland, is used to examine large and highly radioactive samples. In situations where the user facility is at a licensed nuclear site the logistics of examining active components are greatly simplified because of pre-existing experience of transportation and handling issues.

Diffraction contrast tomography allows a map to be made of the grains and their boundaries by correlation of the grain image in the x-ray transmitted beam and the diffracted Laue spot image of the grain. This tool has been used to map grain growth, grain boundary modification, recrystallization, and phase changes. With micro-tomography on the same sample, one can superimpose the image of a propagating crack on the grain map to find out which grain boundaries are selected for crack growth. This is an immensely powerful tool for characterizing defect distributions, voids, and bubbles on the scale of the microstructure.

Phase contrast tomography is based on changes in refractive index and, therefore, phase at boundaries between regions of a sample with different densities. The technique often provides images in situations for which transmission based tomography is insensitive. The method has achieved successes in biology but has not yet been applied to activated materials.

4.3 Scattering

Small-angle scattering using x-rays and neutrons is a common approach for studies of inhomogeneities covering size ranges from Å up to 10 μm (using ultra small-angle techniques). Small-angle neutron scattering measurements (SANS) and ultra-small-angle scattering have measured the distribution of voids and defects in oxide dispersion strengthened steels by using the magnetic cross-section of the material to separate the non-magnetic defect scattering from other inhomogeneities in the steel in an unambiguous way. SANS has been also used to map creep cavitation (see Figure 4) in reactor pressure vessel steels close to the toe of a weld. (See "Quantification of creep cavitation damage around a crack in a stainless steel pressure vessel," P.J. Bouchard, P.J. Withers, S.A. McDonald and R.K. Heenan, Acta Mater, 52 2004 23–34.) There is now overlap (and agreement) between reciprocal space scattering methods of sizing particles and real space tomography in the 1–10 μm range.

Neutron diffraction measurements of residual stress have been made since the 1980s but the application to irradiated material is relatively new. Measurements of stresses in irradiated welds with 20,000 R/hr on contact have been carried out at the Chalk River Nuclear Laboratories of Atomic Energy of Canada by staff at the National Research Council of Canada. The welds were contained in specially designed containers such that the activity outside the container was less than 4 mr/hr and, therefore, safe to deploy on the reactor main floor. The results, interestingly, showed a systematic reduction of residual stress with damage. Diffraction measurements of irradiated low-enriched uranium molybdenum fuel (75 R/hr on contact) have also been made in shielded containers. The results showed that new phases appeared after irradiation plus an amorphous scattering component. A straightforward extension of the method would be to scan the fuel elements to find the distribution of phases as a function of position from the centre to the cladding generated by the temperature distribution from center to core.

Microdiffraction permits stress measurements to be made as a function of position within grains by the use of small x-ray beams generated by Baez-Kirkpatrick mirrors, with gauge volumes of order 0.4×0.6×0.7 µm3. This allows the stresses to be mapped out across grains, around defects and dislocations, and towards the grain boundaries. This is another powerful tool with good prospects of early application to understanding the stresses around bubbles and the genesis of cracks and crack fronts.

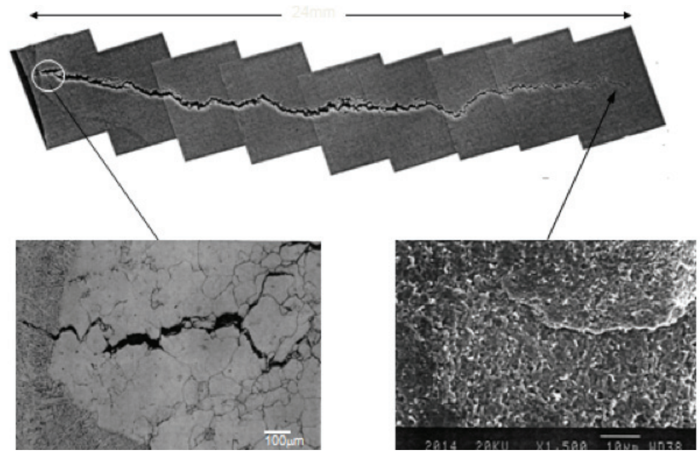


Figure 4. Cavities nucleate and join to form a life-limiting crack (after Phil Withers)

4.4 APS User Survey

In November 2008, a survey gathered input on the needs of scientists planning to use synchrotron x-rays for examining activated materials in connection with the MR-CAT project at the Advanced Photon Source. Figure 5 shows the technique requirements that were identified in the survey. Small-angle, ultra-small angle, and diffuse scattering were considered important for examining defects. (See Diffuse X-ray scattering measurements of point defects and clusters in iron," R.E. Stoller, F.J. Walker, E.D. Specht, D.M. Nicholson, R.I Barabash, P. Zscack, and G. Ice, J. Nucl. Mater, 367-370 2007 p. 209.) Both diffraction with mm size beams to measure phases and microdiffraction techniques to obtain stress distributions on the grain scale were identified. Surface diffraction and grazing incidence small angle scattering to examine near surface effects and corrosion were considered necessary. Tomography, radiography, phase contrast, and fluorescence imaging were all identified as highly important. XAFS, micro-EXAFS and x-ray photoemission were considered to be highly valuable spectroscopies.

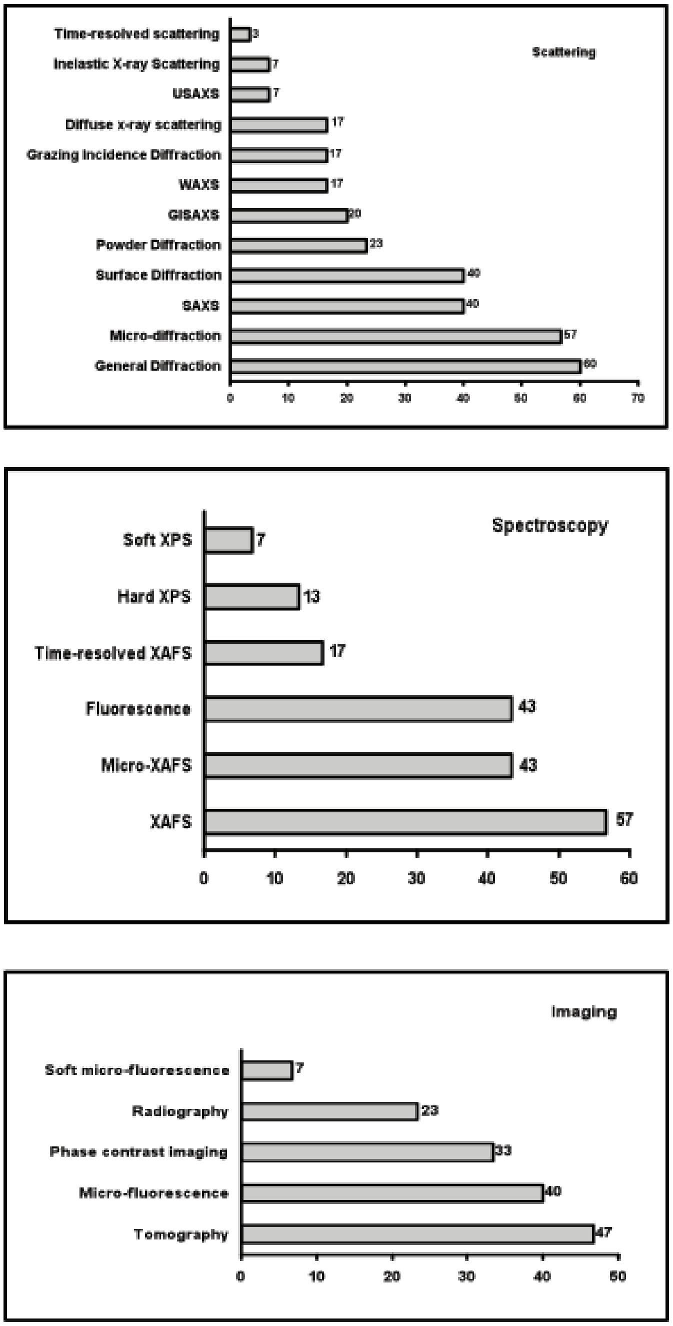


Figure 5. APS User Survey: Technique requirements (after MeiMei Li)

The beam size requirements were divided nearly equally between sizes less than 0.1 µm2, between 0.1 and 1.0µm2, and large beams for bulk measurements. Control over a wide range of temperatures was desired. 500–1000°C corresponds to high temperature reactors and fusion first walls. Temperatures between room temperature and 500°C correspond to thermal reactors; and cryogenic temperatures are sometimes used to minimize thermal phonon scattering when using diffuse scattering. Mechanical facilities included the ability to carry out in situ crack initiation and growth (stress and imaging) measurements, tension, compression and fatigue and internal pressurization. Structural alloys (30%) comprise the largest group of likely topics followed by nuclear fuels (20%), transmutation products and actinides (20%) and SiC, oxides, nitrides, and carbides (TRISO fuels) (30%). Requirements for specimen preparation, mounting, polishing, and cutting were also identified.

5. New Tools of Decadal Scope

5.1 Requirements in a Decadal Future Facility

The new x-ray facilities currently under development (e.g., the free-electron laser) exceed the brightness of current sources by a wide margin so the likelihood of being able to probe even smaller volumes over smaller time intervals than at present is inevitable.

On the second day of the workshop, the participants considered what capabilities might be required in a facility that could juxtapose irradiation capability with advanced probes a decade in the future. Although there was little consensus on what it would take for science-based certification to supersede the existing “cook and look” paradigm, there was nevertheless consensus that greater application of the neutron and x-ray sources for characterization would be valuable. The capability to irradiate under a variety of conditions (fission reactors, ion beams, spallation sources, etc.) was considered essential. Material test reactors, spallation sources, and ion beams were all discussed.

Regardless of the irradiation source, desirable functionality in a decadal future facility included in situ and ex situ measurements of a range of phenomena, e.g., diffraction during loading; examination of individual grains in activated samples; handling and characterization of "large" components; in situ creep properties with helium; defect kinetics measurements with resolution than overlap with models and characterization of spent fuel. Engineering requirements will likely be dominated by need for insight, particularly under high burn-up conditions, on new fuel types such as Triso, MOX, or Thoria, of which we have comparatively little experience.

5.2 Common Scientific Needs

Breakthroughs in understanding in the next 10 years are likely to come from the coupling between theory and modeling with experiment. At the experimental level, this might call for designing experiments over the whole range of length scales and time scales with input from the modelers. At the facilities level, this calls for complementary techniques, such as irradiation facilities, modern examination methods using high intensity x-rays and neutrons, the means to handle the active materials, mechanical testing and chemistry-related activities, and the input of theorists and modelers. The Materials research collaborative access team at the advanced photon source illustrates the value of complementary techniques brought to bear on a single sample and well supported by theory.

The major point here is that the new non-destructive methods of examination can establish the bulk behavior and the development of the accumulating damage over time. Previous methods provided a highly local view but not the statistical behavior nor the time development. From the point of view of developing models of the defect structure, the time development is vital. We have a theoretical picture of the formation of the initial cascade and the clustering that ensues over a short span of time based on kinetic Monte Carlo methods (See "Kinetic Monte Carlo modeling of cascade aging and damage accumulation in Fe-Cu alloys," P.R. Monasterio, B.D. Wirth, and G.R. Odette, J. Nucl. Mater, 361 2007 p. 12.) However, how the clustering leads to a distribution of voids in space and in time, and how these diffuse through the structure to develop a steady-state spatial distribution is not known. This covers a much longer timescale and needs to be followed. Since similar processes operate throughout the bulk, they may be followed with repeated x-ray and neutron diffuse scattering methods which sample the bulk in a statistical fashion.

For UO2 fuels, a thermal neutron environment is required since this is the situation in present day reactors. One can envisage making repeated ex situ measurements on the same fuel pin after intervals of irradiation to observe the development of the voids and bubbles with time. One can also envisage making repeated x-ray and neutron diffraction measurements of the phase content of the pin as a function of distance from the center of the pin and as a function of time. One may be able to make diffraction contrast tomography measurements to find the grain morphology and examine recrystallization as a function of radial position and time if the gamma background problem can be circumvented. The question of in situ examination of fuel by x-rays within the radiation source has been advocated. Since penetrations into a spallation irradiation source could be made following the development of new phases under irradiation with high energy x-ray diffraction is within the realm of possibility. Likewise, it may be possible to examine TRISO fuels, although this requires a neutron environment tailored to resemble a fast reactor. Repeated measurements taken in situ and ex situ of irradiated fuel would be feasible using tomography, diffraction to identify new phases and their distribution and grain configuration changes. These measurements would show the development over time of the voids and bubbles within the fuel and their distribution. Diffuse scattering would be difficult because of the inhomogeneous content of the element as well as its cylindrical shape.

It is desirable to do fatigue measurements within the radiation environment. Apart from the effect of embrittlement on crack growth, if the time scale for relaxation matches the time scale for fatigue, there will be a marked effect of one upon the other. Likewise, it is important for fast reactors, where the operating temperature is higher than thermal reactors, to determine whether high temperature creep affects the fatigue properties of structural components. There is a remote possibility that the high temperature creep may anneal out the effect of fatigue. Fatigue measurements require penetrations into the radiation source to transmit the cyclic force to the sample, radiation insensitive strain gauges and a fair amount of space but are possible. It would be relatively easy to take the fatigued and irradiated samples from the neutron source and use new tools to look for void clustering and crack growth or make measurements of stress around the crack tip to enable the theory of crack growth. For new materials, the same considerations apply: the need to apply tensile and cyclic loads under irradiation and at a variety of temperatures to follow the creep-fatigue interaction. This would then be followed by examination by the new non-destructive tools.

5.3 User Facility Considerations

With the tools at hand, the easiest measurements to implement address length scales close to that of the microstructure (1–100 μm) where the time developments are slower. Many microstructural level measurements are of direct engineering relevance. The faster length scales having to do with the physics of cascade formation are far harder to access with neutron irradiation than ion bombardment.

Thus, the basic experimental building blocks are a neutron irradiation facility, and the possibility of in situ examination by high energy x-rays. No less important are handling facilities enabling tensile testing and fatigue testing of engineering samples in hot cells, the ability to employ all the methods using x-rays from an intense source on highly active samples, and easy access to neutron scattering tools also adapted for highly active samples. One also needs to have all the classical post-irradiation microscopy, such as electron microscopy. For example, interpreting diffuse scattering data can be problematic if there are several sources of scattering since the particular kind of defect cannot be identified without complementary examination.

An extensive program of irradiation at the Los Alamos Neutron Science Center (LANSCE) was carried out during the Accelerator Production of Tritium (APT) program. Irradiation with a spallation source adds a high energy tail to the neutron spectrum compared with a fission reactor. In addition the production of He4 from α-particles is a couple of hundred times higher than at a reactor. In this respect the spallation spectrum begins to approximate to a fusion spectrum. Many structural materials, steels and Al alloys were irradiated and archived. A pulsed fast neutron source, the Materials Test Station, MTS, could be incorporated in the LANSCE accelerator complex. This would have an average intensity that is twice as high as the ATR with intensity in the pulse 10000 times higher. On the other hand the neutron spectrum has a high energy component compared with a thermal reactor spectrum and the He4 content is about 100 times higher. Both the high energy tail and the He4 modify the response of materials.

One challenge for a new facility is rapid impact and one of the problems with neutron irradiation facilities is their relatively slow damage rate. Thus there is a need for accelerated testing. A very important part of the solution for accelerated testing is ion beam bombardment, which can simulate neutron damage. Ion beam irradiation sources provide damage rates that are typically several orders of magnitude faster than any conceivable neutron irradiation facility. Although they lack the penetration, fission fragments and helium production in prototypic neutron irradiations ion irradiation can serve as a powerful complement. By choosing the bombarding ion, one can simulate self-ion damage (say Fe and Cr in stainless steel) or fission gas accumulation. In a triple beam facility, material is bombarded with heavy ions, as well as H- ions and He4. Synergistic effects of the three beams on damage have been recognized. In the MR-CAT project at the Advanced Photon Source (APS) the intent is to bombard foil samples in a triple beam and examine the damage in situ with a synchrotron beam. Mechanical testing of foil samples is feasible although scaling up to engineering test samples and larger components is not assured.

For real space, spatial resolution neutron techniques cannot compete with x-rays, but because of the variability of elemental scattering lengths, neutrons do provide a way to distinguish elements close together in the periodic table or light elements in the presence of heavy elements. Moreover the magnetic cross-section provides another tool in some cases. The other major advantage of neutron diffraction and scattering is that it is not so difficult to shield against the gamma background from the irradiated materials as tests on fuels and welds have shown. (See Neutron powder diffraction of radioactive low enrichment uranium-molybdenum nuclear fuel," L.M. D. Cranswick, K.T. Conlon, R.L. Donaberger, J. Fox, L. McEwan, R. Rogge, D. Sears, D. Sediako, I.P. Swainson, and T. Whan, European Powder Diffraction Conference, Geneva 2006.) Residual stresses in irradiated components will continue to be relevant to the regulatory process. Dedicated spectrometers for neutron experiments, making use of SANS, diffraction, and stress measurements on irradiated samples will be an important part of a new facility.

6. Cross-Cutting Themes

6.1 Modeling

The needs of the modelers serve to define the requirements for experiments at each length scale. At the atomic level, what are the length and time scales for the fundamental cascades generating the damage? What are the length and time scales for these to come to equilibrium in the material? How are these entities situated in the microstructure and how do they interact with intrinsic defects, such as the grain boundaries, which arguably act as sinks. In turn, the microstructural information is needed to calculate the macroscopic properties of the fuel, such as thermal conductivity, temperature, and swelling.

From the current perspective of modeling, the mathematical description of the microstructure (length scale 1.0 to 100 μm) is recognized as the critical link between the behavior at the atomic level and the behavior of the material at the macroscopic level (see Figure 6). In fact, this is also true of the understanding of the mechanical properties of materials in general. At the atomic level, Kinetic Monte Carlo methods can describe the distributions

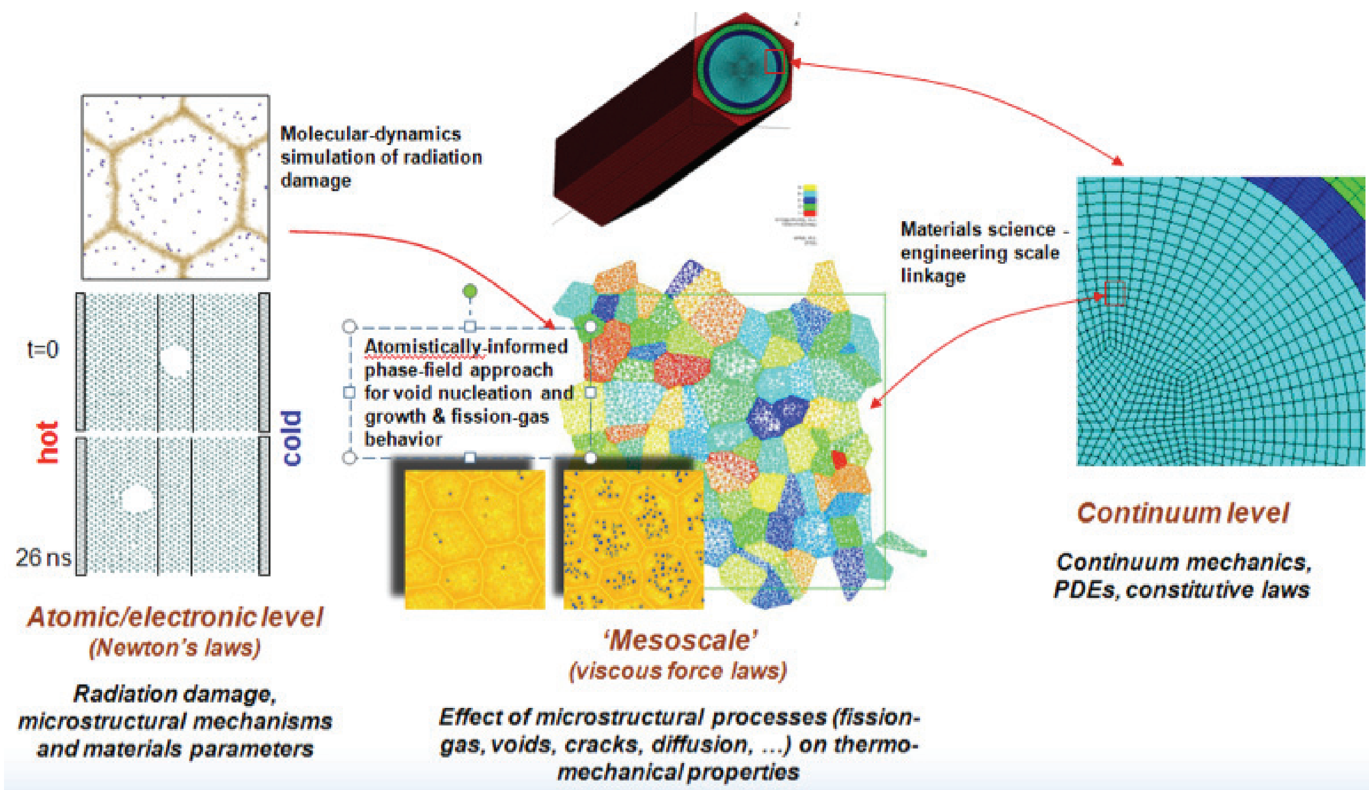


Figure 6. Hierarchical multi-scale approach to nuclear fuel simulation (after Dieter Wolf)

of vacancies and interstitials immediately after (30 fs) a fission product cascade has been initiated. In the following 20 ps, these vacancies and interstitials are thought to coalesce into small clusters over a spatial volume of diameter about 3 nm (0.003 µm). The outstanding fundamental proviso is that the interatomic potential for U and its 5f electrons remains unsatisfactory.

How voids and clusters populate the grains of fuel material is currently addressed by the “phase-field” model, which replaces every grain boundary by an order parameter varying continuously from 0 to 1. (See Phase field model calculation of grains and voids in fuel,” S.Y. Hu et al., J. Nucl. Matter, 392 2009 292–300). The model can describe the genesis of gas bubbles and how they are distributed through the grains, in particular the experimentally observed denuded zones close to grain boundaries where there are fewer bubbles (see Figure 7). With this description of the inhomogeneities within the grains, a constitutive law for the local thermal conductivity can be derived. In turn, finite element models of behavior on the macroscopic length scale use this information to calculate the average thermal conductivity as a function of position and hence derive temperature distributions through the fuel. This is one example of the direction of in which models could be developed. While the modeling effort is close to connecting over the whole length scale, the field is not so much limited by computing power as by descriptive algorithms to describe behavior at the microstructure length scale. Another success has been the microstructural evolution of Fe0.9%Cu after irradiation. The initial vacancy-Cu clusters on a 10Å scale and their coalescence into a series of Cu precipitates have been predicted.

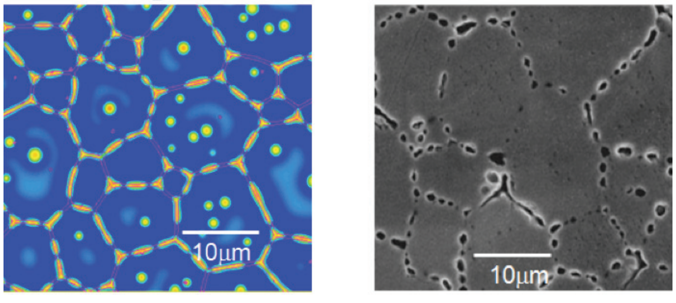


Figure 7. (Left) Phase field calculation using empirical free energy model (after SY Hu et al) & (Right) Irradiated UO2 in pressurized water reactor (after Zacharie et. al)

Currently, many models are not hardware-limited, but algorithm-limited in developing physically-validated methodologies for predicting, for example, microstructural evolution or tensile stress-strain behavior using mesoscale (dislocation dynamics) models. For this reason, much debate focused on measurements pertinent to atomistic and molecular dynamics calculations which were considered most in need of validation. A second theme in the workshop discussions pertained to the engagement of theorists. There was some belief that, even with new validation opportunities, the promise implicit in new generations of models would not be realized without complementary investment in what was called a “virtual computational end station.” Essentially this theme advocated significant investment in developing the computational infrastructure necessary for the community to take advantage of insights that could be achieved by bringing light and neutron source characterizations to bear on activated materials.

6.2 Engagement with the Nuclear Power Industry

There was some discussion at the workshop concerning an assertion that the nuclear power utilities do not believe there are technical challenges for coping with irradiated material but that better allocation of funds is needed to support present examination methods. This suggests that there is currently a mismatch between what is seen as important within the nuclear industry with respect to post-irradiation examination and what researchers outside industry perceive. This mismatch should be explored. Another suggestion was the alignment of goals of research workers outside the industry with the research topics identified by EPRI in its interactions with the industry.

Three themes arose during the breakout discussions which merit attention in a discussion of advanced measurement capabilities. First, some difficulty in engaging nuclear in the advocacy of long term research was noted. The general assumption was that short-term commercial imperatives focused industry attention on the immediacy of existing light water reactor re-certifications. The participants spent some time discussing this issue with solutions ranging from more aggressive participation of the research community at EPRI and NRC meetings; education of the user communities on the way safety case certification occurs now; definition of a DOE champion; efforts to bridge the gaps between the different modus operandi of the DOE offices of Science and Nuclear Energy; and addressing the issue of conflict of interest and intellectual property issues.

7. Conclusion

The overall impact of the workshop was captured in the final panel session. Panelists were asked to name experiments that could be done in the next five years with present resources that would advance the field significantly and those experiments that would have a major impact over the next decade. The panelists were also asked to identify the facilities needed. Two panelists represented academic research and their emphasis was on materials research with irradiated material. Others spoke from the point of view of re-licensing and regulation, requirements for fusion research, and the outlook for the modeling community. Each made suggestions that corresponded to a major program of work.

In materials science research, five priorities were identified for the next five years: 1. the in situ crystallographic response to applied stress in archived irradiated materials; 2. crack growth under fatigue conditions in irradiated zirconium alloys containing hydrides, since this is a major failure route for tubes and cladding; 3. SANS and SAXS measurements on Fe9%Cr steels to identify when deformation-induced voids begin to form around M23C6 particles within the steel and x-ray measurements of strain response to applied stress with microbeams around these precipitates; and 4. synchrotron x-ray measurements to find why materials subjected to hardening by irradiation fail at the same true stress.

On the decadal time scale the following program areas were identified: 1. examination of materials for Generation 4 reactors under irradiation including zirconium alloys and ceramics; 2. fatigue testing at high temperatures to quantify the fatigue-irradiation-creep interaction; and 3. examination of individual particles of M23C6 within the grains of Fe9%Cr steels to see how these interact under deformation with and without irradiation. In the 10-year timeframe, it would be desirable to have a materials irradiation source and associated handling facilities allowing active samples to be subjected to specialized x-ray and neutron tests all on the same site.

From the point of view of re-licensing reactors, the immediate emphasis is on the development of confidence in residual stress accuracy through a program of duplicating measurements at multiple laboratories with the available techniques, neutron and x-ray diffraction, deep hole drilling and the contour method. In the next 10 years, the major goals should be the following: 1. large section stress measurements on irradiated components to find the effect of fluence on weld residual stresses; 2. solution of emerging materials property issues such as the effect of irradiation on the thermal aging of cast stainless steel and austenitic stainless steels; 3. development of NDE techniques to measure radiation damage; and 4. high fluence toughness measurements, including crack growth and stress constraint change with crack growth. On the decadal timeframe, the new facility needs to be able to handle a variety of activated samples of different sizes and irradiated intensities and needs to be flexible enough to handle emerging issues. High temperature property development and characterization of irradiated materials should have a high priority.

For fusion reactors, an important task for the next five years is the analysis of oxide dispersion strengthened (ODS) steels with precipitates in the size range of 2–5 nm, which show promise for excellent creep strength and radiation resistance.

However, problems of the variability of the properties with batch need to be addressed. For the next decade, a major goal for fusion programs will be measurement of high temperature creep properties of ODS steels in a radiation environment in the presence of He^4.

From the perspective of modeling and simulation, in the next five years it should be possible to compute and measure the dynamics of cascade evolution on a picosecond time scale with the aid of synchrotron x-rays, and irradiation with ion beams. The calculation requires massive computing capability, viz. access to petaflop computers. It should be possible in this timeframe to include material specificity in modeling the behavior of cascades. On a 10-year time scale, there is an experimental requirement to match the spatial resolution of the computed cascade to verify the fluctuations within the cascade. An important goal for further benchmarking is obtaining real space and reciprocal space information on identical materials on the same length scale. Finally, since the theoretical effort needed is major, a community of scientists, not isolated groups, is needed to work on the problems.

Several general issues were addressed in the final wrap-up of the workshop's panel discussion. If the global renaissance of nuclear power is to become a reality, then low-cost, accessible, high intensity fast neutron sources must be built, since the availability of irradiation facilities is worse than ever before . The irradiation sources could be either reactor or accelerator based. At the radiation facility there should be access to all the current diffraction, spectroscopic and electron microscope techniques and flexibility to incorporate future developments. Modeling on the mesoscopic scale, with benchmarking by experiment, has enormous potential to connect with engineering behavior. There is no room for exclusive fields of endeavor such as engineering versus science, macroscopic versus mesoscopic, experiment versus modeling; these are all vital constituents of the final common goal.

At the workshop, the materials science requirements for activated materials were articulated. The tools developed over the past decade for examination of materials at the grain level were reviewed. An attempt was made to match the needs to the available tools, making note of the hurdles to be overcome. Finally a set of challenging experiments was suggested that will advance the field in the next five- and 10-year periods with the help of new facilities put in place over this time span.

If the promise of the so-called nuclear renaissance is to be realized, especially in the United States, the breadth and depth of the nuclear science and engineering community must be enhanced substantially. In particular, there is a need to revitalize the materials science of radiation damage. It was clear to workshop participants that x-ray and neutron sources at national user facilities have an important role to play in this endeavor. Further, in addition to cultural changes that would allow the full exploitation of currently available tools and techniques, new capabilities need to be developed if science-based certification is to play a role in the resurgence of nuclear energy. Finally, given the magnitude and urgency of the need for carbon-neutral energy, approaches must be found to reduce the time and cost associated with licensing and certification.

[1]Fast-neutron sources in the United States. The Advanced Test Reactor (ATR) at Idaho National Laboratory is a user facility for neutron irradiation and subsequent examination. It offers a very high flux of fast neutrons (5×10^{14}n/cm^2/s), new post-irradiation and rabbit facilities, and a TRIGA reactor for neutron radiography. The ATR functions as the centerpiece of an academic and industrial group and is also associated with the MR-CAT facility at the APS. The high flux reactor (HFIR) at Oak Ridge offers two locations for fast-neutron irradiation. An extensive program of irradiation at LANSCE was carried out during the Accelerator Production of Tritium (APT) program. Irradiation with a spallation source adds a high energy tail to the neutron spectrum compared with a fission reactor. In addition, the production of He^4 from α-particles is a couple of hundred times higher than at a reactor. In this respect, the spallation spectrum begins to approximate to a fusion spectrum. Many structural materials, steels, and Al alloys were irradiated and archived.

Appendix A: Workshop Agenda

Workshop Title: Research Needs and Opportunities for Characterization of Activated Samples at X-Ray and Neutron User Facilities

Sunday 20 September 2009			
5:30 p.m.	Registration		Tesuque B, C
6:30 pm	Welcome	Tom Holden (NST)	Tesuque B, C
6:40 pm	Overview of DOE Workshops	Mike Fluss (LLNL)	Tesuque B, C
7:00 pm	MaRIE, a proposed signature facility	John Sarrao (LANL)	Tesuque B, C
7:30 pm	Free time		
Monday 21 September 2009			
7:30 am	Continental breakfast		Portal
8:00 am	Workshop goal	Tom Holden (NST)	Tesuque A,B
8:05 am	Workshop philosophy & Rationale	Mark Bourke (LANL)	Tesuque A,B
Session 1:- Measurement needs for activated samples – customer focus **Chair/Scribe Rick Holt / Peter Hosemann**			
8:30 am	Developing advanced nuclear structural materials for use in high radiation environments: An Australian perspective	Lyndon Edwards (ANSTO)	Tesuque A,B
8:55 am	Microstructure Modeling of Nuclear Fuel	Dieter Wolf (INL)	Tesuque A,B
9:20 am	Welding residual stress and material property measurements on irradiated materials	David Rudland (NRC)	Tesuque A,B
9:45 am	Breakout # 1 objectives	Jack Shlachter (LANL)	Tesuque A,B

10:00 am	Refreshments		Portal
Breakout # 1 -- In parallel			
Subgroup 1 Regulatory & Licensing *Chair / Scribe James Wall / Heather Volz*			
10:15 am	Seed Talk	Tiangan Lian (EPRI)	Tesuque A
10:25 am	Address breakout #1 charge	Subgroup	Tesuque A
Subgroup 2 Industry *Chair/Scribe Phil Withers / Don Brown*			
10:15 am	Seed Talk	Rick Holt (Queens University)	Tesuque B
10:25 am	Address breakout #1 charge	Subgroup	Tesuque B
Subgroup 3 Modeling *Chair/Scribe Brian Wirth / Turab Lookman*			
10:15 am	Seed Talk	Malcolm Stocks (ORNL)	Tesuque C
10:25 am	Address breakout #1 charge	Subgroup	Tesuque C
12:00	Lunch		
12:30 pm	Research on Transuranium Systems: Present Capabilities with Large Central Facilities (Lunchtime talk)	Gerry Lander (ITU retd.)	
1:30 pm	Breakout #1 chair summaries		Tesuque B,C
Session 2 Ongoing measurements on activated samples ***Chair/Scribe Angus Lawson / Heather Hawkins***			
2:00 pm	Neutron, Synchrotron & Laboratory X-ray imaging of Active Samples	Phillip Withers (UMIST)	Tesuque B,C
2:25 pm	Radiation Damage Effects in Structural Materials using Swiss Spallation Neutron Source and Light Source Facilities	Yong Dai (PSI)	Tesuque B,C
2:50 pm	Radiation Damage Studies at Los Alamos Neutron Science Center (LANSCE) ; What We Did and What we would Still Like to Do	Walt Sommer (LANL Retd.)	Tesuque B,C

3:15 pm	Breakout # 2 objectives	Jack Shlachter (LANL)	
3:20 pm	Refreshments		
Breakout # 2 --- In parallel			
Subgroup 1 Length scales - Nanometers (defects) *Chair / Scribe Mike Nastasi / Thomas Proffen*			
3:40 pm	Seed Talk	Jim Stubbins (UUC)	
3:50 pm	Address breakout #2 charge	Subgroup	
Subgroup 2 Length scale - Microns (Microstructure) *Chair / scribe Gene Ice / Steve Conradson*			
3:40 pm	Seed Talk	MeiMei Li (ANL)	
3:50 pm	Address breakout #2 charge	Subgroup	
Subgroup 3 Length scale – mm (components) *Chair / Scribe Jeff Terry / Don Brown*			
3:40 pm	Seed Talk	Ron Rogge (Chalk River Can.)	
3:50 pm	Address breakout #2 charge	Subgroup	
6:00 pm	Free Time		
7:00 pm	Dinner		
8:00 pm	Material & Fuel Testing for Advanced Nuclear Systems (After dinner talk)	Paul Lisowski (DOE Office of Nuclear Energy)	
Tuesday 22 September 2009			
7:30 am	Continental breakfast		
8:00 am	Breakout #2 chair summaries		

Session 3: New tools and new facilities decadal scope ***Chair/Scribe Mike Fluss/ Ming Tang***			
8:30 am	Materials By Design	Marius Stan (LANL)	Tesuque B,C
8:55 am	Light source opportunities	Gene Ice (ORNL)	Tesuque B,C
9:20 am	Facility needs	Rick Kurtz (PNNL)	
9:45 am	In situ measurements in a radiation environment	Mark Bourke (LANL)	Tesuque B,C
9:55 am	Breakout # 3 objectives	Jack Shlachter (LANL)	Tesuque B,C
10:00 am	Refreshments		
Breakout #3 in parallel			
Subgroup 1 structural components *Chair / Scribe Randy Nanstad / Tiangan Lian*			
10:20 am	Seed Talk	James Wall (EPRI)	Tesuque A
10:30 am	Address breakout #3 charge	Subgroup	Tesuque A
Subgroup 2 Fuels and Waste *Chair / Scribe Ken McClellan / Rex Hjelm*			
10:20 am	Seed Talk	Akos Horvath (HAS KFKI)	Tesuque B
10:30 am	Address breakout #3 charge	Subgroup	Tesuque B
Subgroup 3 Fundamental Materials *Chair/Scribe Alan Hurd / MeiMei Li*			
10:20 am	Seed Talk	Brian Wirth (UCB)	Tesuque C
10:30 am	Address breakout #3 charge	Subgroup	Tesuque C
12:00 pm	Lunch		
12:30 pm	Advanced Test Reactor national scientific user facility (Lunchtime talk)	Mitch Meyer (INL)	
1:30 pm	Breakout #3 chair summaries		

2:00 pm	Panel discussion	Rick Holt (Queen's Univ.) Jim Stubbins (UUC) David Rudland (NRC) Rick Kurtz (PNNL) Dieter Wolf (INL)	
3:00 pm	Open discussion	All	
4:00 pm	Refreshments		
Session 5: Synthesize Draft Conclusions ***Chair Mark Bourke***			
4:20	Draft Workshop Conclusions	Lyndon Edwards (ANSTO)	
5:00 pm	Written input	Tom Holden (NST)	
5:30 pm	Close		
7:00 pm	Dinner		
8:00 pm	Nonproliferation considerations for nuclear energy and nuclear weapons (after dinner talk)	DV Rao (LANL)	

Appendix B: Participants and Readers

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